

This Page Is Inserted by IFW Operations
and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

**As rescanning documents *will not* correct images,
please do not report the images to the
Image Problem Mailbox.**

19



Europäisches Patentamt
European Patent Office
Office européen des brevets

11 Publication number:

0 168 102
A2

— (S)

12

EUROPEAN PATENT APPLICATION

21 Application number: 85201034.7

51 Int. Cl.4: C 11 D 1/72
C 11 D 3/12, C 11 D 3/39

22 Date of filing: 28.06.85

30 Priority: 06.07.84 DE 3424987

71 Applicant: UNILEVER PLC
Unilever House Blackfriars P.O. Box 68
London EC4P 4BQ(GB)

43 Date of publication of application:
15.01.86 Bulletin 86/3

84 Designated Contracting States:
GB

84 Designated Contracting States:
AT BE CH DE FR GB IT LI NL SE

72 Inventor: Czempik, Klaus
Carl-Hesse-Strasse 44
D-2810 Verden(DE)

71 Applicant: UNILEVER NV
Burgemeester's Jacobplein 1 P.O. Box 760
NL-3000 DK Rotterdam(NL)

72 Inventor: Stremmel, Gerd
Bahnhofstrasse 37
D-6831 Brühl(DE)

84 Designated Contracting States:
BE CH DE FR IT LI NL SE AT

74 Representative: Van Gent, Jan Paulus et al.
Unilever N.V. Patent Division P.O. Box 137
NL-3130 AC Vlaardingen(NL)

54 A process for the preparation of a powder detergent composition of high bulk density.

57 The invention relates to a process for the preparation of a nonionic surfactant-containing powder detergent composition wherein a spray-dried sodium sulphate-free base powder containing a portion of the nonionic surfactant is obtained and the remaining portion of the nonionic surfactant is in part sprayed onto the base powder and in part added subsequently in a form obtained by granulation with a carrier containing a mixture of zeolite and alkali metal borate monohydrate and wherein the other detergent components are added subsequently to the base powder. A detergent composition prepared in this manner has a high bulk density despite its high AD content.

EP 0 168 102 A2

A PROCESS FOR THE PREPARATION OF A POWDER DETERGENT

COMPOSITION OF HIGH BULK DENSITY

The invention relates to a process for the preparation of
5 a powder detergent composition of high bulk density and
more particularly to a process for the preparation of a
detergent composition of the aforesaid kind having a high
AD content (AD= active detergent).

10 Powdered detergent compositions are usually manufactured
in a spray-drying process. In such a process an aqueous
slurry of all the spray-dryable detergent components is
spray-dried and to the powder thus obtained, often
referred to as the base powder, there are subsequently
15 added other desired detergent components that are not
spray-dryable such as enzymes, bleaches and the like.
These other components are often added to the base powder
in a granulated form, or if liquid, they are sprayed
onto the base powder or the finished powder mixture.

20 The powder detergent compositions thereby obtained
generally have a bulk density of less than 0.60 kg/l.
The bulk density is essentially dependent upon the bulk
densities of the base powder and additives and is chiefly
25 determined by the composition of the base powder. If for
example the AD content of the slurry is increased, the
base powder obtained will have a low bulk density, as
will the finished powder.

30 Accordingly it is the aim of the invention to prepare by
the afore-described process a finished powder, having a
high AD content in base powder form and a high bulk
density in the finished form as well as all the other
benefits of spray-dried detergent powders such as
35 solubility, wetting and sinking properties.

It has been surprisingly found that this aim can be
achieved by preparing a base powder that is substantially
free of sodium sulphate and in a subsequent step

introducing the remaining detergent components by means of spraying and incorporation in granulate form.

From German Auslegeschrift No. 1,951,556 there is known a process for the preparation of powder detergents wherein a base powder is obtained by spray-drying, the remaining detergent components being subsequently added to the base powder. For example if nonionics are required in the finished detergent, it is advantageous if only a portion of the nonionic material is incorporated in the base powder by spray-drying, the remaining portion being subsequently sprayed onto the base powder and/or added thereto in granulate form together with the other components of the detergent composition. However, this art proposes improving the powder properties of the base powder by incorporating non-surfactant non-hygroscopic substances such as for example sodium sulphate. The non-surfactant components can constitute up to 20% of the base powder.

It has now been surprisingly found in accordance with the invention that when sodium sulphate is largely omitted there results a base powder which has a high bulk density despite its high AD content. Furthermore, it has been found that if a portion of the remaining nonionic surfactant is sprayed onto the base powder and the rest added in a granulated form obtained with the aid of a carrier containing zeolite and perborate monohydrate, a high bulk density finished powder is obtained.

Accordingly therefore the present invention relates to a process for the preparation of a nonionic surfactant-containing powder detergent composition of high AD content and high bulk density, wherein a spray-dried base powder containing a portion of the nonionic surfactant is obtained and the remaining portion of the

nonionic surfactant is in part sprayed onto the base powder and in part added subsequently in a form obtained by granulation with a carrier and wherein the other detergent components are added subsequently to the base 5 powder, which process is characterized in that the base powder is essentially free of sodium sulphate and the carrier for the nonionic surfactant chiefly contains a mixture of zeolite and perborate monohydrate.

10 It is also within the scope of the invention to spray AD combinations of nonionic and anionic surfactants onto the base powder and the carrier mixture.

15 Useful nonionic surfactants can be chosen from nonionics based on natural or synthetic fatty alcohols or oxo alcohols, fatty acid amides and fatty acid alkylolamides.

20 The hydrophobic moiety of the aforesaid nonionic surfactants will generally have a chain length of C_{10} - C_{20} and a degree of ethoxylation of from 5 to 20 mol EO (= ethylene oxide).

25 Preferably, C_{12} - C_{15} alcohols having a degree of ethoxylation of from 5 to 12 mol EO are used.

30 The anionics can be selected from conventional anionic surfactants such as alkylbenzene sulphonates, alkyl sulphates, alkyl sulphonates, alkyl ether sulphates and the like. Preferably, linear alkylbenzene sulphonates, alkyl sulphonates and/or alkyl sulphates or mixtures thereof are used.

The invention will now be described in greater detail.

35

The detergent composition contains one or more active

detergent materials which can be of any known type. It contains at least one nonionic surfactant but can additionally contain other surfactants such as anionic, cationic, and zwitterionic surfactants and mixtures 5 thereof. Preferably the detergent contains a mixture of nonionic and anionic surfactants.

Generally, the content of nonionic surfactant in the finished detergent powder is from 5 to 15% by weight and 10 the content of anionic surfactant from nil to 12% by weight.

Only a portion of the nonionic surfactant can be introduced into the base powder by spray-drying. This 15 portion can be more than one-half of the total quantity of the nonionic surfactant present in the finished powder, but generally the nonionic content of the base powder will not be more than one-half of the total quantity of nonionic material in the finished powder. The 20 remaining portion of nonionic surfactant is in part sprayed onto the base powder; generally, not more than one-half of the remaining portion of nonionic surfactant is sprayed onto the base powder. The other portion is added to the base powder in granulated form with aid of a 25 carrier.

The other surfactants can be completely incorporated in the base powder by spray-drying, or alternatively a portion thereof together with a portion of nonionic 30 surfactant can be sprayed onto the base powder or onto the carrier mixture of the granulate. This procedure is advantageous if a finished powder with a high anionic AD content is required.

35 The base powder further contains known spray-dryable detergent components such as builder salts, silicates,

brighteners, soil-suspending agents and the like. In this connection it is useful if the powder contains a certain amount, for example a few percent, of a zeolite to facilitate incorporation of the sprayed-on nonionic or 5 nonionic/anionic material.

As was stated hereinabove, a portion of the nonionic surfactant is added to the base powder in a form obtained by granulation with a carrier. The carrier contains a 10 mixture of zeolite and perborate monohydrate. The zeolite can be any suitable sodium aluminium silicate that is known for use as a detergency builder in detergent compositions. A useful example thereof is the zeolite HAB A 40 available commercially from Degussa. Sodium 15 perborate monohydrate is especially useful as the perborate monohydrate. The carrier mixture is granulated with the nonionic surfactant or with the nonionic/anionic surfactant mixture; other components such as sodium tripolyphosphate or sodium sulphate can be added to the 20 granulate. The remaining detergent components are subsequently added to the base powder in an appropriate manner, for example in the form of a granulate or powder or liquid. Typical examples of such additives include 25 granulated bleach activators, bleaches, sequestering agents, enzymes, foam depressants, perfumes and the like.

The detergent composition can additionally contain other usual components that are desired for particular reasons 30 in a detergent composition.

The invention will now be described in further detail in the Examples following hereinbelow.

35 Example 1

The following base powder was prepared by conventionally

spray-drying an aqueous slurry comprising:

	<u>Parts by weight</u>
Sodium alkylbenzene sulphonate	3.00
5 C ₁₂ -C ₁₅ oxo alcohol, condensed with 12 mole EO	5.85
Nonylphenol, condensed with 5/9 mole EO (ex zeolite)	0.15
Sodium tripolyphosphate	25.00
10 Sodium silicate, neutral	5.30
Zeolite, HAB A 40	5.00
Sodium CMC	1.06
EDTA	0.27
Brightener	0.187
15 Salts	0.8
Water	8.653

	58.57

20 The bulk density of this base powder was less than 0.60 kg/l.

The base powder was sprayed with 4 parts by weight of a C₁₂-C₁₅ oxo alcohol condensed with 7 moles of ethylene oxide and the bulk density of the product thus obtained was 0.62 kg/l.

A granulate containing the following components was prepared in a granulator:

	<u>Parts by weight</u>
Sodium perborate monohydrate	8.50
Sodium tripolyphosphate	7.00
Sodium sulphate	2.24
35 Zeolite, HAB 40 (80%)	3.00
C ₁₂ -C ₁₅ oxo alcohol, condensed with 7 mol EO	4.00
Water	1.00

This granulate had a bulk density of 0.96 kg/l.

The base powder and the granulate were mixed together and the remaining detergent components were added, namely 8.5 parts TAED granulate (65%), enzyme granules, perfume and a calcium salt of ethylenediaminetetramethylenephosphonic acid. The finished powder had a bulk density of 0.68 kg/l. and an AD content of 14 parts by weight of nonionic surfactant and 3 parts by weight anionic surfactant.

10

Example 2

The following detergent was prepared in a similar manner as in Example 1:

15

<u>Base powder</u>	<u>Parts by weight</u>
Sodium alkylbenzene sulphonate	5.00
$C_{12}-C_{15}$ oxo alcohol, condensed with 7 mol EO	4.00
20 Nonylphenol, condensed with 5/9 mol EO	0.15
Sodium CMC	1.06
Acrylic acid/maleic anhydride copolymer	2.00
Sodium tripolyphosphate	25.00
Zeolite	5.00
25 Sodium silicate ($Na_2O:SiO_2 = 1:3.3$)	5.30
EDTA	0.265
Brightener	0.527
Salts	1.106
Water	8.50

30

The bulk density was 0.60 kg/l.

The base powder was sprayed with a mixture of 4 parts by weight of a $C_{12}-C_{15}$ oxo alcohol condensed with 7 moles ethylene oxide and 1 part by weight sodium alkylbenzene sulphonate.

The bulk density of the powder thus obtained was 0.63 kg/l.

To this powder there was added a granulate of the
5 following composition:

	<u>Parts by weight</u>
Sodium perborate monohydrate	8.58
Sodium tripolyphosphate	4.50
10 Sodium sulphate	5.252
Zeolite, 80%	4.00
Monostearyl phosphate	1.00
C ₁₂ -C ₁₅ oxo alcohol, condensed with 7 mol EO	2.85

15

Thereafter the following components were added:

	<u>Parts by weight</u>
Perfume	0.254
20 TAED granulate (65%)	8.46
Ethylenediaminetetramethylene- phosphonic acid, Ca salt	2.60
Proteolytic enzyme granulate	0.846

25 The finished powder had a bulk density of 0.70 kg/l. and an AD content of 10 parts by weight of nonionic surfactant and 6 parts by weight anionic surfactant.

When in this Example the sodium alkylbenzene sulphonate
30 content of the base powder was increased by 2% and the nonionics content accordingly reduced by 2%, a finished powder having a bulk density of 0.68 kg/l. was obtained.

Claims

1. A process for the preparation of a nonionic surfactant-containing powder detergent composition having a high AD content and a high bulk density, wherein a spray-dried base powder containing a portion of the nonionic surfactant is obtained and the remaining portion of the nonionic surfactant is in part sprayed onto the base powder and in part added subsequently in a form obtained by granulation with a carrier and wherein the other detergent components are subsequently added to the base powder, the base powder being essentially free of sodium sulphate and the carrier for the nonionic surfactant comprising a mixture of zeolite and alkali metal perborate monohydrate.

2. A process according to claim 1 wherein the detergent composition comprises a mixture of nonionic and anionic surfactants.

3. A process according to claim 1 or 2 wherein the base powder contains zeolites.

4. A process according to claims 1 to 3 wherein the base powder contains less than one-half of the total quantity of nonionic surfactant and not more than one-half of the remaining portion of the total quantity of nonionic surfactant is sprayed onto the base powder.

5. A process according to claims 1 to 4, wherein a portion of an anionic surfactant is sprayed onto the base powder together with the nonionic surfactant.

6. A process according to claims 1 to 5 wherein a portion of an anionic surfactant is sprayed together with the nonionic surfactant onto the carrier mixture of the granulate.